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SODIUM OXALATE AS A STANDARD IN VOLUMETRIC ANALYSIS

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SODIUM OXALATE AS A STANDARD IN VOLUMETRIC ANALYSIS

ABSTRACT

In order to bring to date the information on the standardization of solutions by means of sodium oxalate, Bureau of Standards Circular No. 40 (now circular No. 381), Sodium Oxalate as a Standard in Volumetric Analysis, has been revised. This was done in connection with the analysis of the new lot of sodium oxalate No. 40c which is to replace the present sample No. 40b. Some new tests for purity and data on the effect of rapid titration are given.

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I. INTRODUCTION

1. PURPOSE AND SCOPE OF THE CIRCULAR

This circular is issued for the purpose of publishing a summary of the work done at this bureau and elsewhere which has led to the selection of sodium oxalate as a primary standard. This fourth edition has been revised with special reference to the methods employed and the results obtained in the testing of the sodium oxalate which is now issued as standard sample No. 40c.

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2. REASONS FOR ISSUING A PRIMARY STANDARD

No argument is needed to show that the nature and purity of the primary standard employed is fundamental to the accuracy of any volumetric process. In including among the standard analyzed samples substances of high and known purity to serve as primary volumetric standards, this bureau hopes to remove many of the discrepancies in analysis which arise from the use of unsuitable or impure standards. Examination of a number of samples of sodium oxalate, purchased both in the open market and upon specification, has shown that although some of the samples were of satisfactory purity, others, sometimes from the same makers, were far from pure and in some cases bore misleading labels. This bureau has succeeded in obtaining a large amount of sodium oxalate of the requisite purity. This is now issued as standard sample No. 40c, in a bottle containing 60 g, the price of which is $2. The terms and instructions for ordering are given in the supplement to Bureau of Standards Circular No. 25. With each sample is sent a certificate stating the composition of the material and brief directions for its use.

3. CONSIDERATIONS AFFECTING THE CHOICE OF A STANDARD

The criteria for the selection of a substance which may serve both as a primary and a working standard have been generally recognized, being summed up by McBride[1] as follows:

(*a*) There must be reasonable ease of preparation and accurate reproducibility.

(*b*) The purity must be determinable with sufficient accuracy, and the purified materials must be stable under ordinary conditions of the laboratory.

(*c*) The use of the material in regular work must demand neither complex apparatus nor difficult manipulations.

(*d*) Such precision must be obtainable when it is used with ordinary care that one or at the most a very few determinations suffice for the fixing of the value of a standard solution.

(*e*) The accuracy obtainable under ordinary conditions of its use in standardization must be at least as great as that required in the use of the solution to be standardized.

These various factors will be considered with special reference to their application to sodium oxalate, and the reasons which have led to its selection as a primary standard will be outlined.

II. PREPARATION OF SODIUM OXALATE

1. PURIFICATION

In his original paper upon the use of sodium oxalate as a volumetric standard Sörensen[2] recommended two methods of purification, viz, crystallization from water and precipitation from the aqueous solution by alcohol. The latter process is recommended by him especially for the production of a voluminous finely divided precipitate, which shows less tendency toward decrepitation upon heating when used as an acidimetric standard. On the other hand, the alcohol precipitation

[1] J. Am. Chem. Soc., **34**, p. 394; 1912. (See also Sörensen, Z. anal. Chem., **44**, p. 142; 1905.)
[2] Z. anal. Chem., **36**, p. 639; 1897.

has the disadvantage of yielding a product which is slightly more hygroscopic than that crystallized from water (p. 5). Sörensen's observation that two crystallizations from water will usually be sufficient to effect the purification has been confirmed at this bureau. The chief difficulties in the preparation of sodium oxalate on a large scale arise from (*a*) the sparing solubility of the salt (about 3 g in 100 ml of water at 20° C. and 6 g in 100 ml of water at 100° C.) and (*b*) the action of the solution [3] of sodium oxalate upon the containing vessels, especially glass. The latter property is especially objectionable, since there thus results a product containing insoluble matter, such as calcium oxalate and silica, and excess alkali, usually in the form of sodium carbonate or bicarbonate. The question of suitable methods and apparatus for the purification upon a commercial scale was not investigated by this bureau, although the poor quality and lack of uniformity of many of the commercial samples showed the need of such a study on the part of the manufacturers. The fact that under proper conditions sodium oxalate of high purity can be produced on a large scale at reasonable expense justifies the selection of this material for the purpose under consideration.

2. REPRODUCIBILITY

The experience of this bureau has shown that samples of pure sodium oxalate prepared from materials known to be impure and derived from various sources agreed within the limits of accuracy of the titration (about 1 part in 2,000), both among themselves and with such purchased samples as satisfied the test for purity described in the following section.

III. TESTING OF SODIUM OXALATE

1. GENERAL PRINCIPLES

Sörensen's conclusion [4] in regard to the testing of standards is as follows: "The purity of a primary volumetric standard should be investigated by means of qualitative tests, the performance and quantitative value of which must be accurately determined for each particular primary volumetric standard. Each newly prepared portion of a primary volumetric standard must, of course, be subjected to tests for purity." In accordance with this view he devised a number of tests for impurities likely to be present in sodium oxalate.[5] These tests were used in practically the original form, except the tests for neutrality [6] and for iron, which were modified as the result of experiments conducted at this bureau. The following is a brief description of the tests employed and the results obtained with the lot of sodium oxalate now issued by the bureau as a standard:

2. SPECIAL TESTS

(*a*) WATER.—Hygroscopic water, determined by heating 10 g to constant weight at 105° C., amounted to 0.01 per cent.

Loss in weight between 105° and 240° C. amounted to 0.05 per cent. This includes occluded water as well as the products of de-

[3] Blum, J., Am. Chem. Soc., **34**, p. 130; 1912.
[4] Z. anal. Chem., **44**, p. 155; 1905.
[5] Z. anal. Chem., **42**, pp. 333–359, and 513–516, 1903. Merck, "Prüfung der Chemischen Reagenzien auf Reinheit," 1905. Krauch-Merck, "Chemical Reagents," 1907.
[6] Blum, J., Am. Chem. Soc., **34**, p. 136; 1912.

composition of $NaHCO_3$ or $NaHC_2O_4$ as the case may be, or volatile organic impurities. An electric oven was used, since in a gas-heated oven absorption of sulphur compounds from the products of combustion may take place.

(*b*) NEUTRALITY.—Water was freed from carbon dioxide by boiling for some time while passing through it a current of air free from carbon dioxide. Two grams of the salt was dissolved in 150 ml of this water in a quartz flask, and 0.2 ml of phenolphthalein (1 per cent solution in alcohol) added. The solution was boiled for 10 minutes and cooled while passing into it through a quartz tube a current of air free from carbon dioxide. The color of the resulting solution was compared with that of a solution representing 4 per cent transformation of phenolphthalein. Such a solution is prepared as follows: Transfer 10 ml of 0.1 *N* sodium hydroxide and 0.2 ml of phenolphthalein to a volumetric flask, dilute to 100 ml, and mix thoroughly. Then dilute 4 ml of this solution to 150 ml. The latter solution will represent 4 per cent transformation of phenolphthalein.

The test solution and standard must be of the same volume when the comparison of colors is made. Each ml of 0.01 *N* acid or alkali required to titrate the test solution to the standard color indicates the presence of approximately 0.08 per cent of $NaHCO_3$ or 0.06 per cent of $NaHC_2O_4$. (If 4.0 g of sodium oxalate be used, the standard color should be equivalent to 6 per cent transformation of phenolphthalein and each milliliter of 0.01 *N* acid or alkali would represent one-half of the above percentages of $NaHCO_3$ or $NaHC_2O_4$.) By simple colorimetric comparison it was found possible to estimate the departure from neutrality within 0.01 per cent if the material was nearly pure and 2.0 g samples were employed. Any pink color of lighter tint than the standard indicates the presence of less than 0.02 per cent of $NaHC_2O_4$. If the colors would exactly match, no $NaHC_2O_4$ would be present. The presence of 0.02 per cent of $NaHC_2O_4$ would just discharge the pink color. A greater percentage of $NaHC_2O_4$ would cause the solution to remain colorless, and would require titration to determine the acidity. Tests of this material produced no pink color, and titration showed the presence of 0.04 per cent of $NaHC_2O_4$.

(*c*) INSOLUBLE MATTER.—This salt gave a clear solution, both before and after ignition to carbonate, and contained no material insoluble in water.

(*d*) SULPHATE.—Ten grams of the salt was ignited to carbonate in a platinum crucible using an electric muffle furnace. If an electric furnace is not available, an alcohol burner may be used to avoid contamination which might result if a gas flame were used. The residue was dissolved in a small volume of water and made faintly acid with hydrochloric acid. The solution was heated to boiling, 5 ml of a 10 per cent solution of barium chloride added, and the solution allowed to stand overnight. Sulphate equivalent to 0.005 per cent of Na_2SO_4 was found.

(*e*) CHLORIDE.—Ten grams of the salt was converted to carbonate by ignition in a platinum crucible, the residue was dissolved in water, acidified with nitric acid, and silver nitrate was added. No precipitate appeared on long standing, but only an opalescence which was found to be equivalent to 0.001 per cent of Cl.

(*f*) OXIDES OF IRON, ALUMINUM, ETC.—Ten grams of the salt, converted to carbonate and dissolved in 100 ml of water, produced

a clear solution. This solution was acidified with hydrochloric acid, methyl red indicator added, and then ammonium hydroxide until the indicator just changed to yellow. No perceptible precipitate appeared after standing on the steam bath for two hours. Tests on synthetic solutions under the same conditions showed that the presence of 0.003 per cent of Fe_2O_3 or Al_2O_3 produces a perceptible precipitate. Although this test is not as sensitive as the thiocyanate test for iron, it is more useful because it is more inclusive. If a careful blank on the reagents is carried along, the filtrate from the sulphate test may be used for this test.

(*g*) Potassium.—Five grams of the salt was converted to carbonate in a platinum crucible and dissolved in a small quantity of water. The solution was neutralized with hydrochloric acid, evaporated to dryness on the steam bath, and the residue dissolved in 15 ml of water. Five ml of a solution of sodium cobaltinitrite was added and the solution allowed to stand overnight. No precipitate appeared. (The solution of sodium cobaltinitrite is prepared by dissolving 25 g of $NaNO_2$ in 50 ml of water and then adding 15 ml of 6 *N* $HC_2H_3O_2$ and 2.5 g of $Co(C_2H_3O_2)_2.4H_2O$ or an equivalent quantity of cobalt nitrate or chloride. The solution is allowed to stand overnight, filtered, and diluted to 100 ml.)

(*h*) Organic Impurity.—One gram of the salt when heated in a recently ignited test tube with 10 ml of concentrated sulphuric acid until the appearance of fumes, produced an insignificant discoloration in the acid.

Of these tests the only one that is not conclusive is the last, since it is very evidently impossible to make a single test which will detect every possible organic impurity. This test was devised by Sörensen, who found that by means of it he could detect organic impurities in oxalic acid, when the organic material nonvolatile at 130° C. amounted to 0.012 per cent, while with 0.042 per cent of such impurity present a decided brown coloration was produced. The purity of our standard sample was confirmed by the agreement of its reducing value, within the experimental limits, with samples prepared from different sources by successive crystallizations and by alcohol precipitations.

IV. STABILITY OF SODIUM OXALATE

1. PRESERVATION IN THE DRY STATE

Observations at this bureau extending over a period of 10 years have shown that dry sodium oxalate does not undergo any appreciable change in composition or properties when preserved in stoppered bottles, even when exposed to the light.

2. HYGROSCOPIC PROPERTIES

Sörensen [7] found that 10 g of his sodium oxalate absorbed only 0.0009 g (0.009 per cent) of water when exposed for three days to the influence of "air over water." Lunge,[8] however, found that two 5 g samples of sodium oxalate, when placed in a desiccator in a "moist atmosphere," absorbed, respectively, 0.0188 g and 0.0252 g of water. Neither of these experiments as described is

[7] Z. anal. Chem., 42, p. 349; 1903.

[8] Z. angew. Chem., 18, p. 1527; 1905.

conclusive; since in neither do we know the humidity to which the salt was exposed. Exposure to air saturated with moisture is not a satisfactory test, not alone because material to be used as a standard would seldom be exposed to an atmosphere of such high humidity, but also because under such conditions a slight lowering in temperature, which is likely to occur in a laboratory at night, will cause actual precipitation of moisture upon the walls of the vessel and, of course, upon the substance being tested. Numerous observations at this bureau have shown that under such conditions substances which are not intrinsically hygroscopic may take up several per cent of moisture. The following method was therefore employed in some unpublished tests made at this bureau by Dr. William Blum. Ten-gram samples of sodium oxalate from various sources were dried at 240° C. and were then exposed in a thin layer to air of known humidities, in desiccators containing sulphuric acid of definite concentration and consequently having known aqueous vapor tension.[9] The tests extended over a period of two months, the samples being weighed at irregular intervals in order to detect any erratic absorption. From these tests it was concluded that material crystallized from water (in very fine crystals) showed a maximum absorption of about 0.01 per cent of water in humidities up to 70 per cent; while samples precipitated by alcohol absorbed 0.04 per cent of water under the same conditions, the greater absorption being due no doubt to the greater surface of such materials. No tests were conducted with humidities above 70 per cent since the atmosphere in a balance room will seldom exceed that humidity. While the results are no doubt dependent upon the exact condition of the material, they indicate the order of magnitude of errors likely to arise from moisture absorbed on occasional exposure of this salt to the atmosphere. However, if the sample has been frequently exposed and the presence of moisture is suspected, the material may readily be redried by heating to 105° C. No appreciable error is likely to arise through absorption of water while a sample for analysis is being weighed on an open watch glass. In general, it may be stated that this salt possesses no intrinsic hygroscopic properties, but only such as are common to all substances in a similar state of division.

3. TEMPERATURE OF DECOMPOSITION

Sörensen [10] recommended the temperature of 240° to 250° C. for drying sodium oxalate, after careful tests in which the loss in weight, residual moisture content, and degree of decomposition were determined. He found that after being heated to this temperature for several hours no more than 0.01 per cent of H_2O remained in the salt and no decomposition had taken place. These experiments have been confirmed at this bureau. Thus in one case a sample of sodium oxalate, previously heated to constant weight at 240° to 250° C., when heated to 300° C. for 72 hours lost only 0.03 per cent of its weight and contained a corresponding amount of sodium carbonate as determined by titration. A temperature of 240° to 250° C. may therefore be recommended as a safe and necessary temperature for thorough initial drying of this salt.

[9] For data on the preparation of such solutions see an article by R. E. Wilson, J. Ind. Eng. Chem., **13**, p. 326; 1921.
[10] Z. anal. Chem., **42**, p. 349; 1903.

4. STABILITY OF STOCK SOLUTIONS

It has been shown [11] that solutions of sodium oxalate do not decompose appreciably upon boiling, but that they readily attack glass, and give rise to a precipitate of calcium oxalate. This latter effect is noticeable to a lesser degree when the solutions are preserved in glass at the ordinary temperature. For this reason, and also because of the possible decomposition of a solution of sodium oxalate by sunlight, the use of a stock solution for standardizing is not recommended.

V. USES OF SODIUM OXALATE

Sodium oxalate has been suggested as a primary standard for both oxidimetry and acidimetry, which uses will be considered separately, having regard to (*a*) the methods and apparatus employed, (*b*) the precision attainable—that is, the reproducibility of results—and (*c*) the absolute accuracy of the results obtained.

1. OXIDIMETRY

(*a*) Conditions for Standardization.—The principle of the standardization of permanganate solutions with sodium oxalate is the titration of the oxalic acid liberated by the action of sulphuric acid upon a weighed amount of the sodium oxalate. No special apparatus is required and the operation is very simple. The process may be represented by the following simple equations:

$$Na_2C_2O_4 + H_2SO_4 = Na_2SO_4 + H_2C_2O_4$$

and

$$3H_2SO_4 + 5H_2C_2O_4 + 2KMnO_4 = K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O$$

From these equations it is evident that one molecule of sodium oxalate is equivalent in reducing power to two atoms of ferrous iron. The factor for the ratio $Na_2C_2O_4$:2Fe is therefore 0.8335, using the 1925 International atomic weights. Similarly the factor for available oxygen in manganese ores, is 0.1194 ($Na_2C_2O_4$:O); while the factor for manganese in the bismuthate method is 0.1640 ($5Na_2C_2O_4$: 2Mn). Other factors may be readily calculated from the appropriate equations, or from the accepted ratios, theoretical or empirical, to the iron values.

While various persons have used and recommended this salt as an oxidimetric standard since it was first proposed by Sörensen, few of them have investigated the exact conditions to be employed. Schillow [12] and Skrabal [13] have investigated the dynamics of the reaction between permanganate and oxalic acid. Schroeder [14] has studied the titration of oxalic acid with permanganate, and McBride [15] has investigated at this bureau the conditions for the standardization of permanganate with sodium oxalate. The latter author found that over a considerable range of conditions the variations were less than 1 part in 1,000.

The present recommended procedure is as follows:

In a 400 ml beaker or Erlenmeyer flask dissolve an accurately weighed sample of 0.25 to 0.30 g of sodium oxalate in 200 to 250 ml

[11] Blum., J. Am. Chem. Soc., **34**, p. 129; **1912**.
[12] Ber., **36**, p. 2735; 1903.
[13] Z. anorg. Chem., **42**, p. 1; 1904.
[14] Z. Öffent. Chem., **16**, p. 270; 1910.
[15] J. Am. Chem. Soc., **34**, p. 393; **1912**.

of hot water (80° to 90° C.), and add 10 ml of sulphuric acid (1:1). Titrate at once with a 0.1 N solution of potassium permanganate, *stirring or shaking the solution vigorously and continuously.* The permanganate must not be added more rapidly than 10 to 15 ml per minute,[16] and the last 0.5 to 1 ml must be added dropwise, with particular care to allow each drop to be fully decolorized before the next is introduced. The excess of permanganate used to cause an end point color must be estimated by matching the color in another beaker or flask containing the same quantity of acid and hot water. The temperature of the solution should not be below 60° C. by the time the end point is reached. For the standardization of more dilute solutions of permanganate the same conditions are recommended except that the initial volume and size of sample are proportionately reduced.[17]

(*b*) Precision.—The precision obtained in the titration under these conditions was limited only by the apparatus employed. By the use of weight burettes agreement of duplicates to 1 part in 2,000 could readily be obtained. The precision with volume burettes may be somewhat less than this, depending upon the accuracy of calibration and the care used in the reading of the volume and in maintaining the temperature of the solution constant. In view of the probable warming of the burette during the titration, the use of weight burettes is recommended whenever a precision greater than 1 part in 1,000 is desired.

(*c*) Absolute Accuracy.—The absolute accuracy of the results can not be assumed to be greater than 1 part in 1,000 until an exhaustive investigation of the whole subject of volumetric standards is made. In such an investigation the effect of conditions upon the results obtained with various primary standards—for example, iron, silver, and iodine—must be determined, and the procedure which leads to results of theoretical accuracy must be decided upon independently for each case, if possible. Then the results obtained by the various methods may be compared. In numerous such comparisons which have been made at this bureau and elsewhere, discrepancies of the order of 1 part in 1000 have been observed; but whether these are real or due to errors in the methods used can not be decided with our present knowledge. Thus, in the analysis of a Sibley iron ore (standard sample No. 27) at the bureau, values found for iron gravimetrically agreed within 1 part in 1,000 with those found volumetrically, using sodium oxalate as the primary standard. Aside from the difficulty in selecting the correct end points, as pointed out by McBride,[18] the uncertainties of the atomic weights involved are often of the same magnitude as the discrepancies in question. Examination of the International atomic weight tables for 1907 and 1925, respectively, shows that of 15 of the elements commonly entering into

[16] If the potassium permanganate is added too rapidly, it will be partially decomposed in the hot solution before it can react with the sodium oxalate. The permanganate tint should pass directly to colorless when it comes in contact with the solution of sodium oxalate, without any intermediate brown coloration. If an intermediate brown color is formed, the consumption of permanganate will be in excess of that which is theoretically required. For example, in titrating 0.4000 g portions of sodium oxalate, 56.45 g of a standard solution of potassium permanganate was consumed when the permanganate was added dropwise with vigorous stirring. Exactly the same quantity of permanganate was consumed when the first 50 g was added at the rate of 25 g (or ml) per minute, provided that the solution was so thoroughly stirred or shaken that no intermediate brown color appeared. However, when the permanganate was added at the rate of 25 g (or ml) per minute with little or no shaking or stirring, with the consequent formation of a brown coloration, 56.61 g of the solution was consumed. This is equivalent to an error of 1 part in 350. Therefore, as a matter of safety, it is recommended that the permanganate be added at the rate of 10 to 15 ml per minute.

[17] Blum, J. Am. Chem. Soc., 34, p. 1387; 1912.

[18] J. Am. Chem. Soc. 34, p. 415; 1912.

the volumetric processes (viz, As, Ca, C, Cl, Cu, I, Fe, Pb, Mn, N, P, K, Ag, Na, and S) 5 have had their atomic weights changed during the intervening 18 years to the extent of at least 1 part in 1,000, that of sodium being changed over 1 part in 500. Until the atomic weights of these common elements are more certainly and accurately known, any claim to an absolute accuracy greater than 1 part in 1,000 is manufestly unjustified. These same considerations apply with equal force to the use of sodium oxalate in acidimetry.

2. ACIDIMETRY

Since no investigation of the effect of conditions upon the use of sodium oxalate as an acidimetric standard has been made at this bureau, it is not advisable to certify it for that purpose. A brief summary of the literature will, however, be given.[19]

The principle of its use in acidimetry depends upon the conversion to carbonate by ignition and subsequent titration of this carbonate (or rather of the total alkali) by means of the acid to be standardized. Sörensen recommends the following method of operation: The weighed sample of the salt is carefully heated in a covered platinum crucible, using an alcohol burner. Decomposition takes place in from 15 to 30 minutes, after which the crucible is partly uncovered and the heating continued until the carbon is burned off and the sodium carbonate begins to melt. The residue in the crucible after cooling is treated in a covered beaker with water and a slight excess of the acid to be standardized. The solution is heated on a water bath to expel the greater part of the carbonic acid and is then transferred to a flask in which, after addition of phenolphthalein, it is boiled during the passage of a current of air free from carbon dioxide. After cooling, the excess of acid is titrated with sodium hydroxide of known ratio to the acid.

In a controversy over the relative merits of sodium oxalate and sodium carbonate as acidimetric standards Lunge criticized the use of the former material on account of the difficulties involved, especially the danger of mechanical losses when it is ignited, and the complicated operations involved when phenolphthalein is used as the indicator. Sörensen, and Sörensen and Anderson showed that no appreciable errors arise even when the substance is decomposed rapidly, but that an alcohol flame must be used to avoid contamination by sulphur. Lunge, on the other hand, used a gas flame and an asbestos shield to prevent contamination and mechanical losses. The net result of their controversy was to the effect that sodium oxalate and sodium carbonate yield results differing by no more than about 1 part in 1,000, a difference which is negligible for practical purposes. In an investigation made at this bureau on the use of benzoic acid as an acidimetric standard, Morey [20] found that values obtained with sodium oxalate under approximately the conditions recommended by Sörensen, effecting the decomposition very slowly, agreed closely with those obtained with a number of other standards.

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[19] Consult Sörensen, Z. anal. Chem. **36**, p. 639, 1897; **42**, pp. 333, 512, 1903; **44**, p. 141, 1905; Sörensen and Anderson Z. anal. Chem., **44**, p. 156, 1905; **45**, p. 217, 1906; Sebelin, Chem.-Ztg., **29**, p. 195, 1904; Lunge, Z. angew. Chem., **17**, p. 195, 1904; **18**, p. 1520, 1905.

[20] J. Am. Chem. Soc., **34**, p. 1927; 1912.

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